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**TECHNICAL REPORT**

# High-Energy-Density LCA-Coupled Structural Energetic Materials for Counter WMD Applications

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**14. ABSTRACT**

The major goals of this research project are to investigate the coupling of Linear Cellular Alloys (LCAs) as casings for reactive (thermite) fillers as high-energy-density structural energetic materials. The specific objectives include performing fundamental studies to: (a) investigate mechanics of dynamic densification and reaction initiation in Ta+Fe2O3 and Ta+Bi2O3 thermite powder mixtures and to (b) design and fabricate LCAs of intent (maraging steel) and reactive (tantalum) metals with cell geometries that provide stress transfer for chemical reaction initiation in the thermite filler and allow controlled fragmentation.

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# CONVERSION TABLE

Conversion Factors for U.S. Customary to metric (SI) units of measurement.

| MULTIPLY → | BY → | TO GET |
|---|---|---|
| TO GET ← | BY ← | DIVIDE |
| angstrom | 1.000 000 x E -10 | meters (m) |
| atmosphere (normal) | 1.013 25 x E +2 | kilo pascal (kPa) |
| bar | 1.000 000 x E +2 | kilo pascal (kPa) |
| barn | 1.000 000 x E -28 | meter$^2$ ($m^2$) |
| British thermal unit (thermochemical) | 1.054 350 x E +3 | joule (J) |
| calorie (thermochemical) | 4.184 000 | joule (J) |
| cal (thermochemical/$cm^2$) | 4.184 000 x E -2 | mega joule/$m^2$ ($MJ/m^2$) |
| curie | 3.700 000 x E +1 | *giga bacquerel (GBq) |
| degree (angle) | 1.745 329 x E -2 | radian (rad) |
| degree Fahrenheit | $t_k = (t°f + 459.67)/1.8$ | degree kelvin (K) |
| electron volt | 1.602 19 x E -19 | joule (J) |
| erg | 1.000 000 x E -7 | joule (J) |
| erg/second | 1.000 000 x E -7 | watt (W) |
| foot | 3.048 000 x E -1 | meter (m) |
| foot-pound-force | 1.355 818 | joule (J) |
| gallon (U.S. liquid) | 3.785 412 x E -3 | meter$^3$ ($m^3$) |
| inch | 2.540 000 x E -2 | meter (m) |
| jerk | 1.000 000 x E +9 | joule (J) |
| joule/kilogram (J/kg) radiation dose absorbed | 1.000 000 | Gray (Gy) |
| kilotons | 4.183 | terajoules |
| kip (1000 lbf) | 4.448 222 x E +3 | newton (N) |
| kip/$inch^2$ (ksi) | 6.894 757 x E +3 | kilo pascal (kPa) |
| ktap | 1.000 000 x E +2 | newton-second/$m^2$ ($N-s/m^2$) |
| micron | 1.000 000 x E -6 | meter (m) |
| mil | 2.540 000 x E -5 | meter (m) |
| mile (international) | 1.609 344 x E +3 | meter (m) |
| ounce | 2.834 952 x E -2 | kilogram (kg) |
| pound-force (lbs avoirdupois) | 4.448 222 | newton (N) |
| pound-force inch | 1.129 848 x E -1 | newton-meter (N-m) |
| pound-force/inch | 1.751 268 x E +2 | newton/meter (N/m) |
| pound-force/$foot^2$ | 4.788 026 x E -2 | kilo pascal (kPa) |
| pound-force/$inch^2$ (psi) | 6.894 757 | kilo pascal (kPa) |
| pound-mass (lbm avoirdupois) | 4.535 924 x E -1 | kilogram (kg) |
| pound-mass-$foot^2$ (moment of inertia) | 4.214 011 x E -2 | kilogram-meter$^2$ ($kg-m^2$) |
| pound-mass/$foot^3$ | 1.601 846 x E +1 | kilogram-meter$^3$ ($kg/m^3$) |
| rad (radiation dose absorbed) | 1.000 000 x E -2 | **Gray (Gy) |
| roentgen | 2.579 760 x E -4 | coulomb/kilogram (C/kg) |
| shake | 1.000 000 x E -8 | second (s) |
| slug | 1.459 390 x E +1 | kilogram (kg) |
| torr (mm Hg, $0^0$ C) | 1.333 22 x E -1 | kilo pascal (kPa) |

*The bacquerel (Bq) is the SI unit of radioactivity; 1 Bq = 1 event/s.

**The Gray (GY) is the SI unit of absorbed radiation.

## HIGH-ENERGY-DENSITY LCA-COUPLED STRUCTURAL ENERGETIC MATERIALS FOR COUNTER WMD APPLICATIONS

The major goals of this program are to investigate the coupling of *Linear Cellular Alloys* (*LCAs)* as casings for *reactive (thermite) fillers* as high-energy-density *structural energetic materials.*

The specific objectives include performing fundamental studies to: (a) investigate mechanics of dynamic densification and reaction initiation in $Ta+Fe_2O_3$ and $Ta+Bi_2O_3$ thermite powder mixtures and to (b) design and fabricate LCAs of inert (maraging steel) and reactive (tantalum) metals with cell geometries that provide stress transfer for chemical reaction initiation in the thermite filler and allow controlled fragmentation. The major activities were broken down into three parts and included (i) ***Studies of shock-compression and impact-initiated reactions in thermite powder mixtures*** based on conducting dynamic compaction experiments on thermite powder mixtures with specific materials characteristics, performing time-resolved uniaxial-strain and uniaxial-stress experiments, and using predictive modeling to determine the densification, shock-compression , and reaction initiation behavior in $Ta+Al_2O_3$ & $Ta+Bi_2O_3$ thermite powder mixtures, (ii) ***Fabrication and determination of properties of maraging steel and tantalum-based LCAs of various cell geometries*** including determining the high-strain-rate mechanical properties of LCAs of different cell designs and characterization of impact-initiated reactions in LCAs filled with thermite mixtures, and (iii) ***scaling-up the fabrication process to make 1" diameter, 12-cell, 25% dense LCA for their possible testing as casings at NSWC***.

### Significant Results

***(i) Studies of shock-compression and impact-initiated reactions in thermite powder mixtures:*** The effort focusing on studies of shock-compression and impact initiation of reactions in Ta-based thermite ($Ta+Fe_2O_3$ and $Ta+Bi_2O_3$) mixtures culminated with the Ph.D dissertation of Dr. Anthony Fredenburg. His dissertation established models that provide prediction of the dynamic densification and shock compression response, while capturing the effects of material inherent properties (such as shock impedance, strength, density, etc.) and configurations (packing density, volumetric distribution, powder morphology, etc.) of reactants. In the case of the equivolumetric $Ta+Fe_2O_3$ powder mixtures, pre-densification results in generating $Fe_2O_3$ as the more dominant matrix phase, while in the case of the equivolumetric $Ta+Bi_2O_3$ powder mixture, Ta is the matrix phase and dominates the densification response. Consequently, the overall mechanics of the shock-compression response of the two types of thermite mixtures is quite different. Interestingly, predictions of the effect of initial packing density however, reveal no effect on the crush-strength. Investigation of the reaction initiation thresholds in $Ta+Fe_2O_3$ and $Ta+Bi_2O_3$ thermite systems under uniaxial-stress rod-on-anvil impact loading illustrate that that $Ta+Bi_2O_3$ mixtures react at lower threshold impact energies, than $Ta+Fe_2O_3$ mixtures, with equivolumetric mixtures in both cases having lower thresholds than stoichiometric mixtures. Initial density also appears to affect the reaction initiation threshold, with 90% dense mixtures reacting at lower threshold impact energy than the 70% dense mixtures.

The results of these studies have been published in the following papers:

- D.A. Fredenburg and N.N. Thadhani, "High-pressure equation of State of Properties of Bismuth Oxide," Journal of Applied Physics, Vol. 110 (2011) 0635101-5
- D.A. Fredenburg and N.N. Thadhani, "On the applicability of the P-alpha and P-lambda models to describe the compaction response of highly heterogeneous powder mixtures," submitted to Journal of Applied Physics, August 2011.
- D.A. Fredenburg, "Shock Compaction and Impact Response of Thermite Powder Mixtures" Ph.D. Dissertation, Georgia Institute of Technology, December 2010.

### *(ii) Fabrication and determination of properties of maraging steel and tantalum-based LCAs of various cell geometries: mechanical deformations and reactivity of filler thermite mixture*

Fabrication of the LCA casing begins with paste preparation. During this step, micron-size oxide powders are thoroughly mixed with binders, lubricants, and a solvent to produce a stiff paste. For the Maraging 200 (M200) steel composition (Fe18Ni12Co4Mo0.7Ti), powders of $Fe_3O_4$, $Ni_2O_3$, $Co_3O_4$, Mo, and $TiH_2$ are compounded with a methocellulose, steric acid, and water extrusion aid system. During shape fabrication, the paste is extruded through a honeycomb die and allowed to air dry. Upon drying, the extruded body is easily handled and is transferred to a furnace for oxide reduction and sintering in a hydrogen atmosphere which transforms the body into the finished metal part. Reduction occurs at about 800°C and sintering at 1300°C. The M200 system provides a high strength, relatively inert platform. To provide a highly reactive, high strength platform, LCAs were also fabricated from tantalum using a completely organic, non-oxidizing extrusion aid system of Acetic Acid tert-Butyl Ester (tert-Butyl Acetate) as the solvent and Ethyl Methacrylate as the binder. The binder system is thermally removed cleanly without contaminating the Ta and sintering of the tantalum extrusion is completed at 1600°C in an argon atmosphere. Successful maraging steel (M200) and tantalum LCAs of about 25% relative density with 8-cell pie and 9-cell waffle geometry, made from maraging steel and tantalum were produced, and are shown in Figure 1.

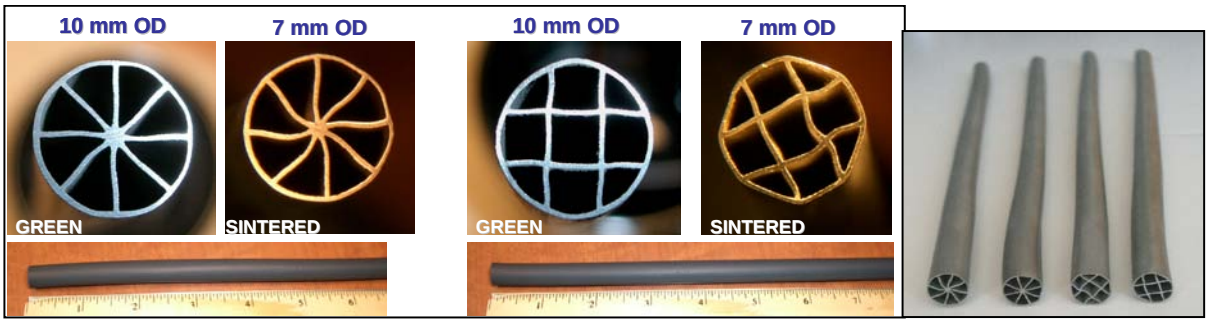


**Fig. 1. Maraging Steel LCAs of 8-cell Pie and 9-Cell waffle design with 250 µm wall thickness, ~7 mm final diameter, and 20 cm length in the final sintered state following sintering of the extruded green.**

The dynamic mechanical behavior of solid maraging steel rods and the 6.8 mm diameter LCAs shown in Figure 1 was investigated using Taylor rod-on-anvil impact tests, by correlating the transient and final deformation profiles recorded by the IMACON 200 camera, with those

predicted via AUTODYN-3D simulations employing the Johnson-Cook (J-C) strength model. The results of this work constituted the MS degree thesis of Adam Jakus who is now pursuing a Ph.D. at the Northwestern University. The impact-initiated chemical reactivity of LCAs of the various designs filled with the $Ta+Bi_2O_3$ thermite mixture was also compared with that of the hollow cylinder of similar overall density filled with the same thermite mixture. The experiments were conducted by impacting the filled LCAs and cylinder against a rigid anvil to determine the effect of confinement and shear stress transfer on the reactivity of the thermite filler. The results showed that the thermite filler in the LCA reacts at impact velocities as low as ~60 m/s, while that with the hollow cylinder reacts at ~108 m/s, thereby indicating that the reactivity of the thermite filler is significantly higher with the LCA, thereby requiring a much lower impact energy threshold for reaction initiation than with the hollow cylinder.

The overall results of these studies are published in the following papers.

- N.N. Thadhani and J.K. Cochran, "Energetic Materials", DTRA Basic and Applied Research Program Newsletter, V2, N3, p. 4-6, July,(2009)
- T. McCoy, A. Fredenburg, A. Jakus, J. Cochran, N.N. Thadhani, "Processing and Dynamic Properties of High-Energy-Density LCA-Coupled Structural Energetic Materials," Proceedings of JANNAF Conference, Dec 8-12, 2009, LA Jolla.
- J.L. Cheng, Y.W. Lee, S.W. Du, N.N.Thadhani, and H.H. Hng, "Kinetic study of thermal and impact-initiated reactions in $Al+Fe_2O_3$ nanothermite mixtures," Combustion and Flame, 157 (2010) 2241-2249.
- A. Jakus, D. A. Fredenburg, and N.N. Thadhani, "High-Strain-Rate Deformation of Maraging Steel Linear Cellular Alloys: Mechanical Deformations," Materials Science & Engineering – A, Accepted for publication, November 2011 (Available online 7 December 2011).
- D. A. Fredenburg, A. Jakus, T. McCoy, J. Cochran, and N.N. Thadhani, "High-Strain-Rate Deformation Behavior of Maraging Steel Linear Cellular Alloys: Energetic Encapsulants," Submitted to Journal of Impact Engineering, November 2011.

***(iii) Scaling-up the fabrication process to make 1" diameter, 12-cell, 25% dense LCA for their possible testing as casings at NSWC.***

Following our initial success with fabricating maraging steel (M200) and tantalum LCAs of 6-cell pie and 9-cell waffle geometries of 6.8 mm final diameter, and 25% relative density, we started work on a new 12-cell 1" diameter design of the LCA. The goal for the conceptual design of this 12-cell 1" diameter LCA illustrated in Figure 2 was to potentially use it for encapsulating energetic mixtures to address the question if segregated reactants are safer and as-reactive as mixed systems, by placing them in different outer compartments and HE in the core, in contrast to a uniform mixture placed either in the LCA of similar design or simply a hollow cylinder. Figure 3 illustrates the design of the 12-cell LCA and hollow cylinder

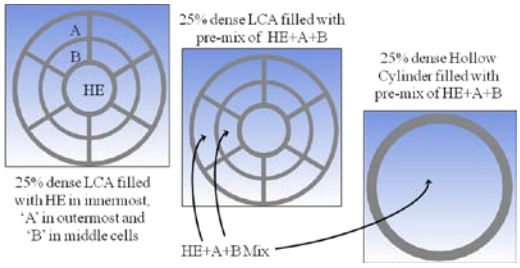


Fig. 2. Concept of LCA with compartments compared to hollow cylinder of same density.

and photos of the extrusion die built for making the 1” diameter LCAs with 12 cells on two

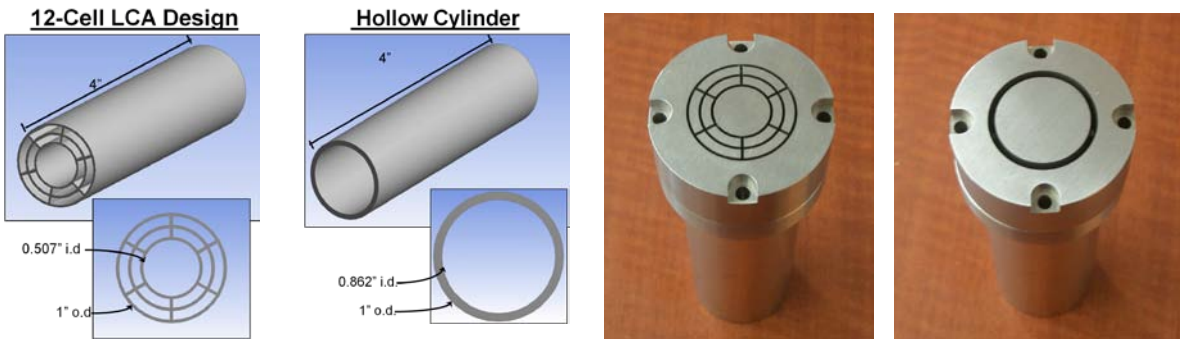


Fig. 3. Design of the 12-cell LCA and hollow cylinder and photos of extrusion die built for making 1” diameter LCAs with 12 cells and hollow cylinder, both of similar 25% density.

concentric outer layers and a center hollow cylinder. A hollow cylinder extrusion die was also produced to provide comparison to the multifunctional die. Dimensions of both are shown in the figure and final extrusions from the 12-cell and the hollow cylinder are targeted to be 25% dense. The extrusion die-land was formed by electrical discharge machining (EDM) of the die-land using W-Cu composite tool that has been made using EDM to form the die-land geometry. Feed holes were drilled on back of the die to provide uniform paste flow to the die-land to ensure complete die-land fill. The die dimensions were designed for 33% shrinkage based on about 10% drying shrinkage and about 25% reduction and sintering shrinkage.

Multiple attempts have been made using the larger dies, for fabricating maraging steel extrusions of about 6 to 8 inches in length. Various controls of extrusion conditions have been investigated to achieve the desired final reductions. Simultaneously, sintering studies have also been performed to reduce the oxides and obtain final metallic LCAs.

Initial work was focused on making key adjustments to the paste configuration, namely controlling the solid and moisture content. For extrusion with the small LCA dies, a low paste solid content of approximately 36% by weight is used to provide pseudoplastic (shear thinning) behavior, meaning that at high rates of shear the material will flow easily or exhibit a low viscosity. For the new larger die, a stiffer paste with higher solids content (~39-47%) is expected to provide better dimensional control. With the small die, pseudoplasticity was present but extrusion pressures were high; as much as 35000 psi. The larger die exhibits pseudoplasticity as well, but with lower pressures. Hence, control of the paste rheology for extrusion through the larger die size becomes even more important variable. Figure 4 shows the general form of the as-extruded 12-cell 25% dense LCA product.


Fig. 4. 12-cell LCA (1.4” dia, 8” lengh) in as-extruded state

The larger size makes it difficult to generate enough shear strain in the paste, with the available plunger size (of 1.7" diameter), to permit controlled flow through the die. The increased number of cells results in reduction of the thickness of the walls, while maintaining the same 25% relative density. A larger extrusion plunger of 2.5" diameter was then fabricated to allow proper flow of material. This however, significantly adds to the volume of the paste to be extruded and associated materials cost, but it did improve the extrusions and we reached the point that good, round extrusions with a length of ~8-10 inches were being fabricated fairly routinely. A range of paste compositions were also investigated varying in solids content from 39-47%. We arrived at the point that consistently good extrusions were achieved with pastes containing ~41% solids. However, after extrusion, the LCA is soft, easily deformed and cannot be handled in any way until it is dry. Thus, it had to be dried in place hanging from the extrusion die. If left to dry in the open, the exterior dries first with the 6% shrinkage and because the interior is soft, it deforms to accommodate the exterior drying shrinkage. As the interior dries, it cracks because the exterior is unyielding, Figure 5. So we spent a good deal of time trying to arrive at a drying schedule to prevent interior cracking. The procedure that has evolved is to wrap the support frame around the extrusion with plastic wrap (Saran Wrap) to completely enclose it. A thermometer and humidity indicator is placed internally in the extrusion process. We heat the wrapped enclosure with an exterior, variable, hot air blower and monitor the interior temperature and humidity. Within an hour, the humidity increases to 100% and the temperature increases by ~6$^{o}$F. The best results were obtained when drying occurred over 5-6 days and those were the conditions for the sample in Figure 6. Obviously, with these drying times, progress was tediously slow. An obvious route to reduce drying time was to increase solids content, and reduce water content in limits that allows good extrusion. Batches with 43 and 44% of solids content were subsequently prepared for testing, with higher solids content resulting in poor extrusions. With the current design, the less than optimally-controlled rheology of the extrusion paste, yields less strength to the thin walls of the LCA which can result in their lack of retaining the shape following extrusion and promotes cracking unless drying is carefully controlled.

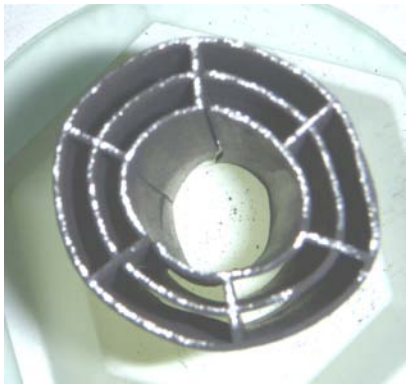

Fig. 5. Interior longitudinal cracks due to rapid drying

Fig. 6. LCA with good straightness and roundness. Two interior circumferential cracks can be seen in the top right photo and there is one small interior longitudinal crack that is not visible in the photo.

An additional problem that occurs with Maraging extrusions during sintering in the ($Ar+H_2$) reducing atmosphere to ~1250$^oC$ to obtain 1" diameter LCAs, is the significant sagging of the walls (Figure 7) with the present 6-spoke design of the 12-cell LCA. The sagging occurs when the LCAs are sintered laying flat in a horizontal tube furnace while being purged with $Ar+H_2$ gas. As seen in Figure 7, the radial webs support the cylindrical shells but bending occurs between the webs. The current 12-cell LCA design does not have enough radial struts to provide appropriate support to the cell walls. The large scale sagging encountered during sintering is due to a combination of this lack of radial support for the LCA walls of large size but small thickness, and also due to the creation of a partial liquid phase at the reducing temperature during sintering.

Fig. 7. Sagging upon sintering in horizontal furnace.

As described, the low axial-stiffness LCA designs (in the absence of radial webs) cannot be reduced and sintered horizontally because at some stage, the structure collapses. Thus, sintering of the LCAs was performed vertically in short lengths due to limited furnace dimensions. This created problems because our tube furnaces require 4" diameter ceramic tubes for upright sintering of the LCA samples, and these large diameter tubes are prone to cracking. So the sintering process has also been difficult. It should be noted that our original 7-mm diameter 8-cell pie and 9-cell waffle LCA energetic capsule designs developed in this program, had a significantly higher axial stiffness (and also a 66% smaller diameter). Hence, the design was stiff enough in the as-extruded state to be handled in the green state and drying was then uniform because air could be passed in a controllable manner through the entire length of the tube. Also, it could be reduced and sintered horizontally in a small diameter tube furnace and as can be seen in Figure 1, long straight lengths (without any sagging) were routinely achieved.

To determine the temperature range in which liquid formation occurs during sintering/reduction, interrupted sinter studies have been carried out to terminate sintering at progressively higher temperature to estimate at which the temperature of slumping occurs for horizontal sintering. It was determined that slumping is occurring at ~ 800$^o$ C which is early in the reduction cycle for the Fe, Co, and Ni oxides. Thus, a liquidus composition for $Fe_3O_4$, $Ni_2O_3$, $Co_3O_4$, has been reached. There is no ternary phase diagram for these three oxides so it is impossible to pinpoint the liquid composition and the total liquid content is limited because total melting does not occur. However, there is just sufficient liquid phase formed does that promotes slumping.

One obvious approach to avoid the liquid formation is to change starting chemistries. Thus, a series of compositions are currently under investigation. Compounding the Maraging steel composition with $Fe_2O_3$ and metal powders of Co, Ni, and Mo, plus $TiH_2$, is in process. It is likely we will avoid liquid formation on reduction/sintering at high temperature and will be able to successfully perform the sintering, for demonstrating this approach by about January 12-16$^{th}$.

## Path Forward

As we move forward, it is also recommend that we consider other designs for the larger size LCA structures to increase the stiffness with both design and relative density. A suggested design is seen in Figure 8, where a series of "W"s replace the middle circle. This should significantly increase stiffness to help maintain roundness and not make filling any more difficult. It is believed that the current composition for Maraging steel would work well with this design because as can be seen from Figure 7, the slumping during horizontal firing occurs between the radial struts. It is thought the triangular support structure would not increase filling difficulty because there are only two geometries of triangles in the outer support region.

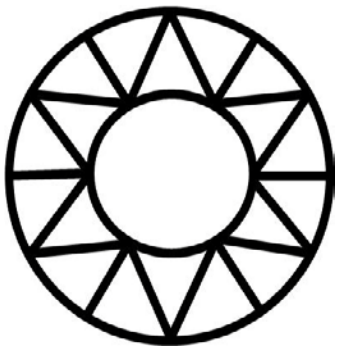

Fig. 8. Suggested LCA energetic capsule design with high stiffness and open internal cell for easy filling

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